

MASTER'S THESIS

Mechanical behavior of polytetrafluoroethylene in tensile loading under different strain rates and creep&relaxation test



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DECLARATION

This thesis is a presentation of my original research work. Wherever contributions of others are involved, every effort is made to indicate this clearly, with due reference to the literature, and acknowledgement of collaborative research and discussions. The thesis was done under the guidance of Doc. Ing. David Herák, Ph.D., at the Czech University Life Sciences of Prague, Faculty of Engineering.

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ABSTRACT

The present work is concerned with the study of the mechanical behavior of polytetrafluoroethylene (PTFE) in tensile tests performed under different strain rates and on creep&stress relaxation tests with using standard specimens. The strains are measured through an universal extensometer. The engineering strain rates ($\dot{\epsilon}'1 = 7,8 \text{ mm/s}$, $\dot{\epsilon}'2 = 20\text{mm/s}$, $\dot{\epsilon}'3 = 27\text{mm/s}$), the constant stress (creep) approximately at 75N and constant strain (stress relaxation) about at 0.00035 were controlled by using the measured elongation as input to the control system of the testing machine. The stress-strain curves are interpreted by comparing effect of different strain rates with the capability of describing complex non-linear mechanical behaviour. And then we observed and evaluated the time-dependence behaviours of PTFE under creep&relaxation tests.

Key Words: Polytetrafluoroethylene, Strain rates, Creep, Relaxation.

ABSTRAKT

Tato práce se zabývá studiem mechanického chování Polytetrafluoroethylenu (PTFE) v tahových zkouškách provedených za různých rychlostí deformace, dotvarováním a relaxací napěťových testů. Vzorky byly měřeny pomocí univerzálního extenzometru. Použité hodnoty vzorků jsou: ($\dot{\epsilon}'_1 = 7,8$ mm / s, $\dot{\epsilon}'_2 = 20$ mm / s, $\dot{\epsilon}'_3 = 27$ mm / s), konstantní napětí :75N a konstantní napětí (dopružování): 0,00035. Testování bylo provedeno pomocí měřeného prodloužení na testovacím zařízení. Křivky napětí-deformace jsou interpretovány porovnáním účinku různých rychlostí deformace s popisem nelineárního mechanického chování. Výsledky byly hodnoceny na základě časové závislosti PTFE po relaxačních testů a testů tečení.

Klíčová Slova: Polytetrafluoroethylenu, Rychlostí Deformace, Tečení, Relaxačních.

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1.INTRODUCTION

In recent years polytetrafluoroethylene (PTFE) has received considerable attention due to its special physical characteristics such as high melting point, very good resistance against chemicals and extremely low friction. The complex nonlinear behaviour of polytetrafluoroethylene remains one of the most severe limitations for its even wider use in the chemical and petrochemical industries.

The combined effect of rate dependency and very large inelastic deformations can be extremely complex. The mechanisms proposed so far to explain microscopically the material behavior are not able to elucidate all aspects of these coupled phenomena. In a certain sense, the material behavior of PTFE can be considered superplastic. A wide class of materials - metals, ceramics, intermetallics, nanocrystalline, polymer, etc - show superplastic behavior under special processing conditions. The most important characteristic of a superplastic material is its high strain rate sensitivity of flow stress that confers a high resistance to neck development and results in the high tensile elongations characteristic of superplastic behavior. Superplastically deformed material in tensile tests gets thinner in a very uniform manner, rather than forming a 'neck' (a local narrowing), which leads to fracture.

Although, up to now, there is no precise physical definition of superplasticity phenomenon in polymeric materials; from a phenomenological point of view, superplasticity can be defined as very high deformations prior to local failure. In the case of tensile tests under controlled strain rate, this means very high elongations of the specimens before rupture. Despite the lack of definition of any fundamental mechanism for superplastic behavior, the evaluation of the influence of strain rate on superplastic properties of a polymer sheet material is a basic requirement for its economical use. This objective is accomplished by the execution of a set of tensile tests at different rates.

The one of purpose of this work is to describe mechanical behavior of PTFE in tensile loading under different strain rates. Another goal is to compare experimental curves and the stress-strain curve under different strain rates with the capability of describing complex nonlinear mechanical behavior. The main contribution of this work is to present a simple but

reliable alternative approach to predict the mechanical behavior of PTFE at different strain rates.

And the another purpose of this work is to describe mechanical behavior of PTFE on stress relaxation and creep tests in tension. The time-dependence of strain may be defined as the 'creep' behavior, and a study of creep, together with the equally important phenomenon of recovery, is essential for a full understanding of the mechanical properties. The stress-strain-time relationship at a constant temperature may be obtained by observing either the strain-time relationship at a constant stress (creep). We know when materials that creep or cold flow are used as gaskets in flanged joints, the phenomenon of stress relaxation is generally encountered. In flanged, bolted connections, parts of PTFE will cold flow between the flange faces with a resultant decrease in bolt pressure. Such relaxation in gasket stock may result in a leaky joint. Tightening the flange bolts during the first day after installation will usually maintain bolting pressure and prevent leakage; thereafter, stress relaxation will be negligible. It is also important to note that the present study is mainly focused on the influence of these tests on the material response in tensile tests performed at room temperature and it is not the goal to discuss the microscopic mechanisms involved.

2. LITERATURE REVIEW

The results presented here are a continuation of a multidisciplinary effort aimed at understanding the mechanical response of a well characterized polymer both from an experimental view point and coupled with creating graphs. The polymer described in this study is poly(tetrafluoroethylene) (PTFE). It was chosen for several reasons, including its use as a common engineering material for small high-performance parts and its availability from several manufacturers. While studied extensively in the past, it has received little attention in the open literature for the last 25 years. Some previous data on the tensile properties of PTFE have been published. A little data on the tensile response of Teflon as a function of temperature and under tensile creep conditions is presented in a DuPont publication [1], but the test conditions are inadequately explained and there is no material pedigree given. By far the best examination of the tensile response of PTFE was undertaken by Koo [2–4]. PTFE specimens have been tested in compression and tension at differing strain rates and temperatures [5,6]. Experimental tests of relaxation in tension of PTFE were developed and the results compared with the prediction of Maxwell’s model [7]. Several constitutive models have been proposed to predict the complex mechanical behavior of thermoplastic and thermoset materials. Strain rate and temperature-dependent behavior of polymers has been described by means of constitutive models [8–14]. Also, some studies were made to report deformation damage in solid polymers [15–18]. PTFE is known to exhibit time and temperature dependent mechanical behavior and display highly nonlinear response during loading and unloading. Khan and Zhang [19] published the strain rate hardening, creep and relaxation behavior of PTFE at room temperature. Rae and Dattelbaum [20] studied the compression stress-strain relationship of PTFE at different temperatures and strain rates. The behavior of filled PTFE has been studied in a uniaxial relaxation test, uniaxial tension tests with different strain rates and retardation tests after unloading (see Kletschkowski et al. [21,22]). The mechanical behavior of polymers under high strain rates has been presented in the literature [23–25]. Some research on the creep properties exists [26–29], but in terms of engineering deformation, only six references have come to the attention of the authors. In 1963, Davies published a paper on the development of a Split-Hopkinson bar system. As part of this report, a single room temperature stress/strain curve for PTFE was presented at $\dot{\epsilon} = 1700 \text{ s}^{-1}$. [30]. The maximum strain imposed in this system was only 3%. Further high strain rate data on Teflon versus temperature was published by Gray [31] and Walley [32]. The effects of temperature on the mechanical response were also briefly discussed. DuPont has published a

guide to material properties that contains three compression curves at 23, 100 and 204,8 °C to approximately 25% strain [33]

3.MATERIAL PROPERTIES

3.1 General Properties of PTFE

PTFE is a polymer discovered in 1938 by Roy Plunkett of the DuPont Company. Its chemical structure consists of chains of carbon atoms bonded together, with branches of fluorine atoms attached. The material is often referred to as polytetrafluoroethylene (PTFE), while Teflon is a registered trademark of the DuPont Company.

PTFE has become an important engineering material. Its major benefits are its non-adhesive character, inertness, resistance to chemical attacks and relatively high strength. Moreover, specific physical properties can be enhanced by adding filler compounds or altering the manufacturing process. Some physical disadvantages that PTFE experiences are high sensitivity to temperature changes and low durability to creep&stress relaxation. The complex and highly non-linear nature of these characteristics presents a delicate task for any engineer who wishes to predict the behavior of PTFE.

Another drawback that PTFE suffers from is its high melt viscosity that makes injection and blow molding impossible, leaving more expensive manufacturing methods, such as sintering and extrusion, the only choices for part production.

3.2 Structure of PTFE

The smallest component of any polymer is called the monomer. In PTFE, the monomer consists of two carbon atoms, each of them having two fluorine atoms attached, Figure 1. When this unit is repeated a long chain is formed and thousands of such chains form the macroscopical structure. Depending on the temperature and the manufacturing method, the chains can exist in an ordered, aligned pattern, known as a crystalline state, or being entangled with a random chain orientation, like cooked spaghetti in a bowl, known as an amorphous state. In fact, both crystalline regions and amorphous regions may exist simultaneously, which is the case in PTFE, which is referred to as a semi crystalline state.

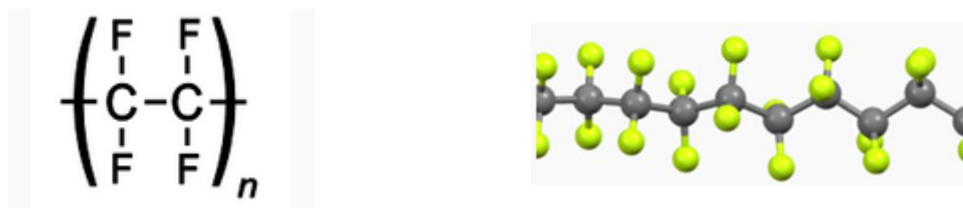


Figure 1. Structure of PTFE

The bonds within each chain are strong covalent bonds. The secondary bonds that act between

two chains are weaker than the covalent bonds, and the larger the distance between two chains, the weaker the secondary bond. In a crystalline region the chains are tightly packed and consequently, the secondary bonds are stronger than in an amorphous region. The degree of crystallization will therefore affect the strength of the polymer. One property that often is of importance for polymers is the glass transition temperature, which is associated with the long-range molecular motions. Below the glass transition temperature the molecules are restricted in motion and consequently very stiff, like a glass. When the temperature is increased above the glass transition temperature a phase transition occurs. Adjacent atoms might move as a unit, which results in a more flexible, leathery structure. Since PTFE is a semi-crystalline polymer this behavior is less emphasized than in a pure amorphous material. The glass transition temperature of PTFE is -97°C .

The flexibility will increase with increased temperature and when the melting temperature is reached, the crystalline bonds are broken apart. By that time, the amorphous regions are already in a liquid state, and the polymer enters a liquid. Differences in chain length and between regions within the polymer make it difficult to define an absolute melting temperature. It is rather defined as a temperature range, and for PTFE it is typically 328°C - 341°C . An interesting property of PTFE is its expansion due to temperature. Most materials expand when exposed to a rise of temperature, which is measured with the linear thermal expansion coefficient. The variation of this coefficient for PTFE is shown in Figure 2. At low temperatures, the expansion increases linearly with the temperature. Around 20°C , a phase transition occurs, which drastically increases the expansion. At high temperatures, the variation is exponentially increasing. The valve is typically assembled below 20°C whereas it operates up to 200°C . The difference in expansion for those situations can be seen in Figure 2.

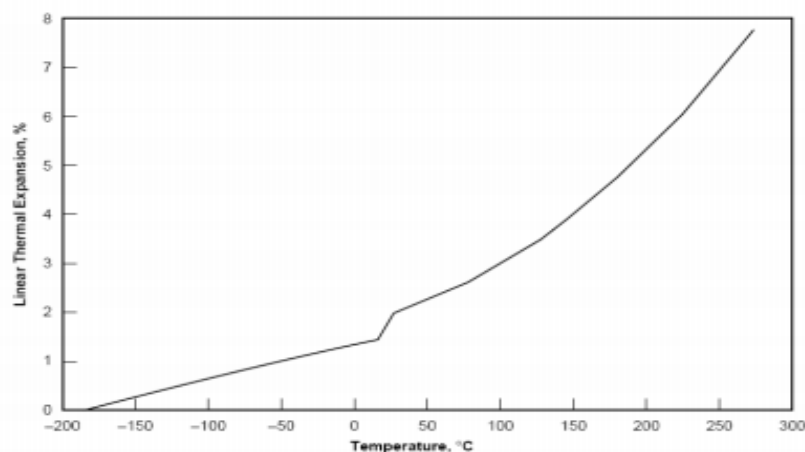


Figure 2. The variation of the linear thermal expansion coefficient with temperature

3.3 Applications of PTFE

The major application of PTFE, consuming about 50% of production, is for wiring in aerospace and computer applications (e.g. hookup wire, coaxial cables). This application exploits the fact that PTFE has excellent dielectric properties.

This is especially true at high radio frequencies, making it suitable for use as an insulator in cables and connector assemblies and as a material for printed circuit boards used at microwave frequencies. Combined with its high melting temperature, this makes it the material of choice as a high-performance substitute for the weaker and lower-melting-point polyethylene commonly used in low-cost applications.

In industrial applications, owing to its low friction, PTFE is used for applications where sliding action of parts is needed: plain bearings, gears, slide plates, etc. In these applications, it performs significantly better than nylon and acetal; it is comparable to ultra-high-molecular-weight polyethylene (UHMWPE). Although UHMWPE is more resistant to wear than PTFE, for these applications, versions of PTFE with mineral oil or molybdenum disulfide embedded as additional lubricants in its matrix are being manufactured. Its extremely high bulk resistivity makes it an ideal material for fabricating long-life electrets, useful devices that are the electrostatic analogues of magnets.

Other:

PTFE (Teflon) is best known for its use in coating non-stick frying pans and other cookware, as it is hydrophobic and possesses fairly high heat resistance.

PTFE is a versatile material that is found in many niche applications:

- It can be stretched to contain small pores of varying sizes and is then placed between fabric layers to make a waterproof, breathable fabric in outdoor apparel.
- It is used widely as a fabric protector to repel stains on formal school-wear, like uniform blazers, in the UK.
- It is used as a film interface patch for sports and medical applications, featuring a pressure-sensitive adhesive backing, which is installed in strategic high friction areas of footwear, insoles, ankle-foot orthosis, and other medical devices to prevent and relieve friction-induced blisters, calluses and foot ulceration.
- Powdered PTFE is used in pyrotechnic compositions as an oxidizer with powdered metals such as aluminium and magnesium. Upon ignition, these mixtures form carbonaceous soot

and corresponding metal fluoride and release large amounts of heat. They are used in infrared decoy flares and as igniters for solid-fuel rocket propellants.

- In optical radiometry, sheets of PTFE are used as measuring heads in spectroradiometers and broadband radiometers (e.g., illuminance meters and UV radiometers) due to PTFE's capability to diffuse a transmitting light nearly perfectly. Moreover, optical properties of PTFE stay constant over a wide range of wavelengths, from UV down to near infrared. In this region, the relation of its regular transmittance to diffuse transmittance is negligibly small, so light transmitted through a diffuser (PTFE sheet) radiates like Lambert's cosine law. Thus PTFE enables cosinusoidal angular response for a detector measuring the power of optical radiation at a surface, e.g. in solar irradiance measurements.
- Certain types of hardened, armor-piercing bullets are coated with PTFE to reduce wear on firearms's rifling that harder projectiles would cause. PTFE itself does not give a projectile an armor-piercing property.
- Its high corrosion resistance makes PTFE useful in laboratory environments, where it is used for lining containers, as a coating for magnetic stirrers, and as tubing for highly corrosive chemicals such as hydrofluoric acid, which will dissolve glass containers. It is used in containers for storing fluoroantimonic acid, a superacid.
- PTFE tubes are used in gas-gas heat exchangers in gas cleaning of waste incinerators. Unit power capacity is typically several megawatts.
- PTFE is widely used as a thread seal tape in plumbing applications, largely replacing paste thread dope.
- PTFE membrane filters are among the most efficient industrial air filters. PTFE-coated filters are often used in dust collection systems to collect particulate matter from air streams in applications involving high temperatures and high particulate loads such as coal-fired power plants, cement production and steel foundries.
- PTFE grafts can be used to bypass stenotic arteries in peripheral vascular disease if a suitable autologous vein graft is not available.
- Many bicycle lubricants contain PTFE and are used on chains and other moving parts.
- PTFE can also be used for dental fillings to isolate the contacts of the anterior tooth so the filling materials will not stick to the adjacent tooth.

3.BACKGROUND

3.1.1 Tension(Tensile) Stress

Tension is a reaction force applied by a stretched string (rope or a similar object) on the objects which stretch it. The direction of the force of tension is parallel to the string, towards the string.

Tensile properties indicate how the material will react to forces being applied in tension. A tensile test is a fundamental mechanical test where a carefully prepared specimen is loaded in a very controlled manner while measuring the applied load and the elongation of the specimen over some distance

Tensile tests are used to determine the modulus of elasticity, elastic limit, elongation, proportional limit, reduction in area, tensile strength, yield point, yield strength and other tensile properties.

The main product of a tensile test is a load versus elongation curve which is then converted into a stress versus strain curve. Since both the engineering stress and the engineering strain are obtained by dividing the load and elongation by constant values (specimen geometry information), the load-elongation curve will have the same shape as the engineering stress-strain curve. The stress-strain curve relates the applied stress to the resulting strain and each material has its own unique stress-strain curve. A typical engineering stress-strain curve is shown below. If the true stress, based on the actual cross-sectional area of the specimen, is used, it is found that the stress-strain curve increases continuously up to fracture.

3.1.2 Linear-Elastic Region and Elastic Constants

As can be seen in the Figure 3, the stress and strain initially increase with a linear relationship. This is the linear-elastic portion of the curve and it indicates that no plastic deformation has occurred. In this region of the curve, when the stress is reduced, the material will return to its original shape. In this linear region, the line obeys the relationship defined as **Hooke's Law** where the ratio of stress to strain is a constant.

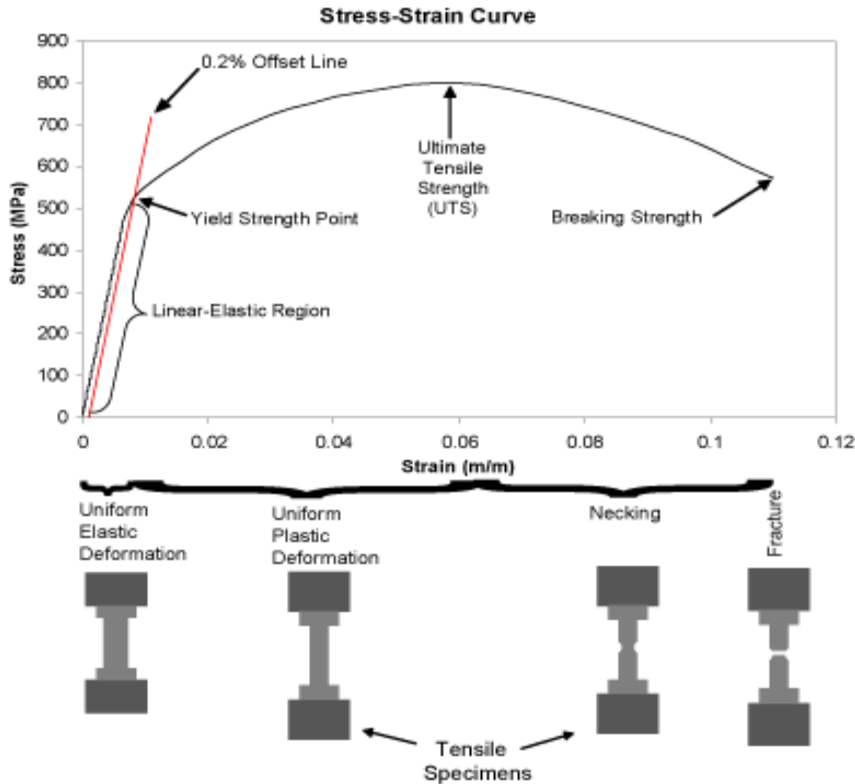


Figure 3.(Stress-Strain Curve)

The slope of the line in this region where stress is proportional to strain and is called the **modulus of elasticity** or **Young's modulus**. The modulus of elasticity (E) defines the properties of a material as it undergoes stress, deforms, and then returns to its original shape after the stress is removed. It is a measure of the **stiffness** of a given material. To compute the modulus of elastic ,simply divide the stress by the strain in the material. Since strain is unitless, the modulus will have the same units as the stress, such as kpi or MPa. The modulus of elasticity applies specifically to the situation of a component being stretched with a tensile force. This modulus is of interest when it is necessary to compute how much a rod or wire stretches under a tensile load.

There are several different kinds of modul depending on the way the material is being stretched, bent, or otherwise distorted. When a component is subjected to pure shear, for instance, a cylindrical bar under torsion, the shear modulus describes the linear-elastic stress-strain relationship.

Axial strain is always accompanied by lateral strains of opposite sign in the two directions mutually perpendicular to the axial strain. Strains that result from an increase in length are designated as positive (+) and those that result in a decrease in length are designated as negative (-). *Poisson's ratio* is defined as the negative of the ratio of the lateral strain to the axial strain for a uniaxial stress state.

$$\nu = - \frac{\epsilon_{lateral}}{\epsilon_{axial}}$$

Poisson's ratio is sometimes also defined as the ratio of the absolute values of lateral and axial strain. This ratio, like strain, is unitless since both strains are unitless. For stresses within the elastic range, this ratio is approximately constant. For a perfectly isotropic elastic material, Poisson's Ratio is 0.25, but for most materials the value lies in the range of 0.28 to 0.33. Generally for steels, Poisson's ratio will have a value of approximately 0.3. This means that if there is one inch per inch of deformation in the direction that stress is applied, there will be 0.3 inches per inch of deformation perpendicular to the direction that force is applied.

Only two of the elastic constants are independent so if two constants are known, the third can be calculated using the following formula:

$$E = 2 (1 + \nu) G.$$

Where: E = modulus of elasticity (Young's modulus)
 n = Poisson's ratio
 G = modulus of rigidity (shear modulus).

A couple of additional elastic constants that may be encountered include the bulk modulus (K), and Lamé's constants (m and l). The bulk modulus is used describe the situation where a piece of material is subjected to a pressure increase on all sides. The relationship between the change in pressure and the resulting strain produced is the bulk modulus. Lamé's constants are derived from modulus of elasticity and Poisson's ratio.

Yield Point

In ductile materials, at some point, the stress-strain curve deviates from the straight-line relationship and Hooke's Law no longer applies as the strain increases faster than the stress. From this point on in the tensile test, some permanent deformation occurs in the specimen and the material is said to react plastically to any further increase in load or stress. The material will not return to its original, unstressed condition when the load is removed. In brittle materials, little or no plastic deformation occurs and the material fractures near the end of the linear-elastic portion of the curve.

With most materials there is a gradual transition from elastic to plastic behavior, and the exact point at which plastic deformation begins to occur is hard to determine. Therefore, various criteria for the initiation of yielding are used depending on the sensitivity of the strain measurements and the intended use of the data. For most engineering design and specification applications, the yield strength is used. The yield strength is defined as the stress required to produce a small, amount of plastic deformation. The offset yield strength is the stress corresponding to the intersection of the stress-strain curve and a line parallel to the elastic part of the curve offset by a specified strain (in the US the offset is typically 0.2% for metals and 2% for plastics).

To determine the yield strength using this offset, the point is found on the strain axis (x-axis) of 0.002, and then a line parallel to the stress-strain line is drawn. This line will intersect the stress-strain line slightly after it begins to curve, and that intersection is defined as the yield strength with a 0.2% offset. A good way of looking at offset yield strength is that after a specimen has been loaded to its 0.2 percent offset yield strength and then unloaded it will be 0.2 percent longer than before the test. Even though the yield strength is meant to represent the exact point at which the material becomes permanently deformed, 0.2% elongation is considered to be a tolerable amount of sacrifice for the ease it creates in defining the yield strength.

Some materials such as gray cast iron or soft copper exhibit essentially no linear-elastic behavior. For these materials the usual practice is to define the yield strength as the stress required to produce some total amount of strain.

True elastic limit is a very low value and is related to the motion of a few hundred dislocations. Micro strain measurements are required to detect strain on order of 2×10^{-6} in/in.

Proportional limit is the highest stress at which stress is directly proportional to strain. It is obtained by observing the deviation from the straight-line portion of the stress-strain curve.

Elastic limit is the greatest stress the material can withstand without any measurable permanent strain remaining on the complete release of load. It is determined using a tedious incremental loading-unloading test procedure. With the sensitivity of strain measurements usually employed in engineering studies (10^{-4} in/in), the elastic limit is greater than the proportional limit. With increasing sensitivity of strain measurement, the value of the elastic limit decreases until it eventually equals the true elastic limit determined from micro strain measurements.

Yield strength is the stress required to produce a small-specified amount of plastic deformation. The yield strength obtained by an offset method is commonly used for engineering purposes because it avoids the practical difficulties of measuring the elastic limit or proportional limit.

Ultimate Tensile Strength(UTS) or, more simply, the tensile strength, is the maximum engineering stress level reached in a tension test. The strength of a material is its ability to withstand external forces without breaking. In brittle materials, the UTS will be at the end of the linear-elastic portion of the stress-strain curve or close to the elastic limit. In ductile materials, the UTS will be well outside of the elastic portion into the plastic portion of the stress-strain curve.

On the stress-strain curve above, the UTS is the highest point where the line is momentarily flat. Since the UTS is based on the engineering stress, it is often not the same as the breaking strength. In ductile materials strain hardening occurs and the stress will continue to increase until fracture occurs, but the engineering stress-strain curve may show a decline in the stress level before fracture occurs. This is the result of engineering stress being based on the original cross-section area and not accounting for the necking that commonly occurs in the test specimen. The UTS may not be completely representative of the highest level of

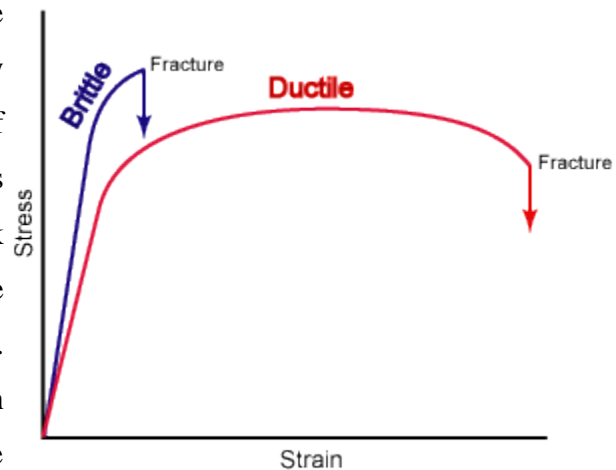
stress that a material can support, but the value is not typically used in the design of components anyway. For ductile metals the current design practice is to use the yield strength for sizing static components. However, since the UTS is easy to determine and quite reproducible, it is useful for the purposes of specifying a material and for quality control purposes. On the other hand, for brittle materials the design of a component may be based on the tensile strength of the material.

3.1.3 Measures of Ductility (Elongation and Reduction of Area)

The ductility of a material is a measure of the extent to which a material will deform before fracture. The amount of ductility is an important factor when considering forming operations such as rolling and extrusion. It also provides an indication of how visible overload damage to a component might become before the component fractures. Ductility is also used a quality control measure to assess the level of impurities and proper processing of a material.

The conventional measures of ductility are the engineering strain at fracture (usually called the elongation) and the reduction of area at fracture. Both of these properties are obtained by fitting the specimen back together after fracture and measuring the change in length and cross-sectional area. Elongation is the change in axial length divided by the original length of the specimen or portion of the specimen. It is expressed as a percentage. Because an appreciable fraction of the plastic deformation will be concentrated in the necked region of the tensile specimen, the value of elongation will depend on the gage length over which the measurement is taken. The smaller the gage length the greater the large localized strain in the necked region will factor into the calculation. Therefore, when reporting values of elongation , the gage length should be given.

Figure 4. Ductility&Brittle comparison



One way to avoid the complication from necking is to base the elongation measurement on the uniform strain out to the point at which necking begins. This works well at times but

some engineering stress-strain curve are often quite flat in the vicinity of maximum loading and it is difficult to precisely establish the strain when necking starts to occur.

Reduction of area is the change in cross-sectional area divided by the original cross-sectional area. This change is measured in the necked down region of the specimen. Like elongation, it is usually expressed as a percentage.

As previously discussed, tension is just one of the way that a material can be loaded. Other ways of loading a material include compression, bending, shear and torsion, and there are a number of standard tests that have been established to characterize how a material performs under these other loading conditions.

3.2 Strain rate

Strain rate is the rate of change in strain (deformation) of a material with respect to time. The strain rate at some point within the material measures the rate at which the distances of adjacent parcels of the material change with time in the neighborhood of that point. It comprises both the rate at which the material is expanding or shrinking (**expansion rate**), and also the rate at which it is being deformed by progressive shearing without changing its volume (**shear rate**). It is zero if these distances do not change, as happens when all particles in some region are moving with the same velocity (same speed and direction) and/or rotating with the same angular velocity, as if that part of the medium were a rigid body.

The strain rate is a concept of materials science and continuum mechanics, that plays an essential role in the physics of fluids and deformable solids. In an isotropic Newtonian fluid, in particular, the viscous stress is a linear function of the rate of strain, defined by two coefficients, one relating to the expansion rate (the bulk viscosity coefficient) and one relating to the shear rate (the "ordinary" viscosity coefficient).

In simple contexts, a single number may suffice to describe the strain, and therefore the strain rate. For example, when a long and uniform rubber band is gradually stretched by pulling at the ends, the strain can be defined as the ratio ϵ between the amount of stretching and the original length of the band:

$$\epsilon(t) = \frac{L(t) - L_0}{L_0}$$

where L_0 is the original length and $L(t)$ its length at each time t . Then the strain rate will be

$$\dot{\epsilon}(t) = \frac{d\epsilon}{dt} = \frac{d}{dt} \left(\frac{L(t) - L_0}{L_0} \right) = \frac{1}{L_0} \frac{dL}{dt}(t) = \frac{v(t)}{L_0}$$

where $v(t)$ is the speed at which the ends are moving away from each other.

The strain is basically the rate of two lengths, so it is a dimensionless quantity (a number that does not depend on the choice of measurement units). Therefore the strain rate is dimensionally the reciprocal of time. In the International System of Units (SI), it is measured in reciprocal of seconds (s^{-1}).

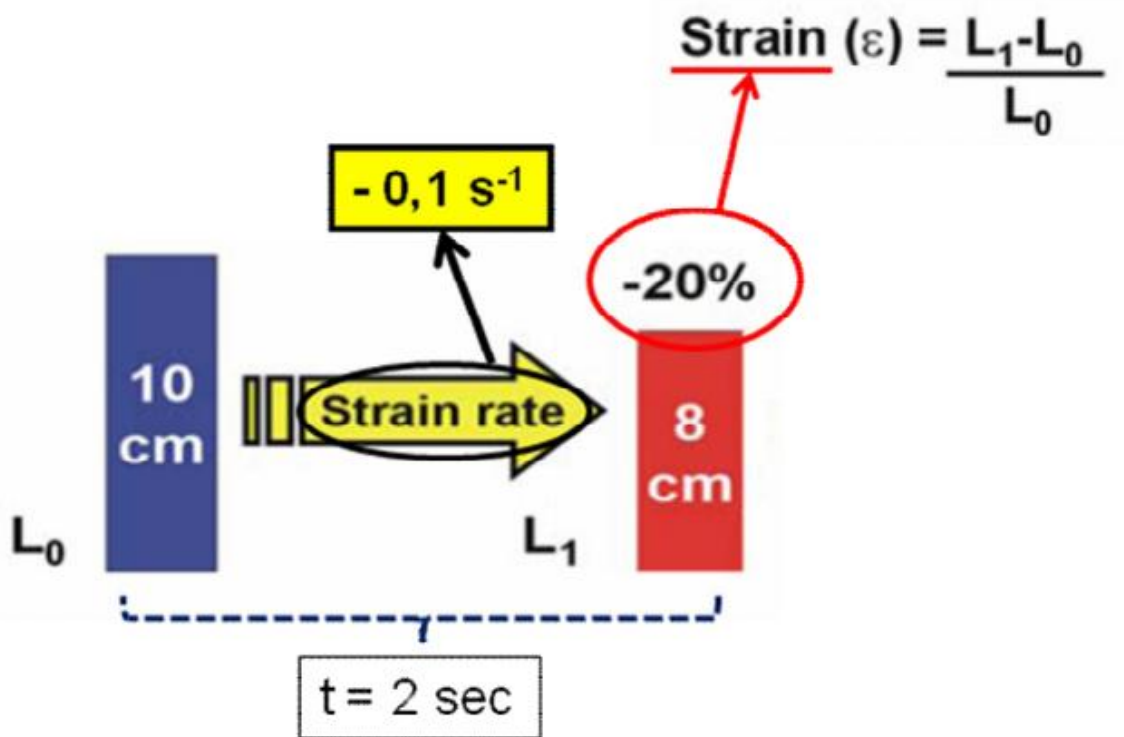


Figure 5. Figure to illustrate the relationship between S and SR. If we assume that this deformation (in this case the shortening) takes 2 seconds, the strain rate is equal to $-0,2/2 \text{ sec}$, which is $-0,1 \text{ s}^{-1}$.

3.3 Creep&Relaxation

Long term performance of polymers

Polymer materials exhibit time dependent behavior. The stress and strain induced when a load is applied are a function of time. In the most general form can be thought of as a 3 dimensional surface. The stress-strain-time relationship, or constitutive law, can be determined by loading a polymer specimen with constant stress (creep) or constant strain (stress relaxation or isometric). We can also construct isochronous curve by cross section of the surface with constant time levels as shown in **Figure 6**.

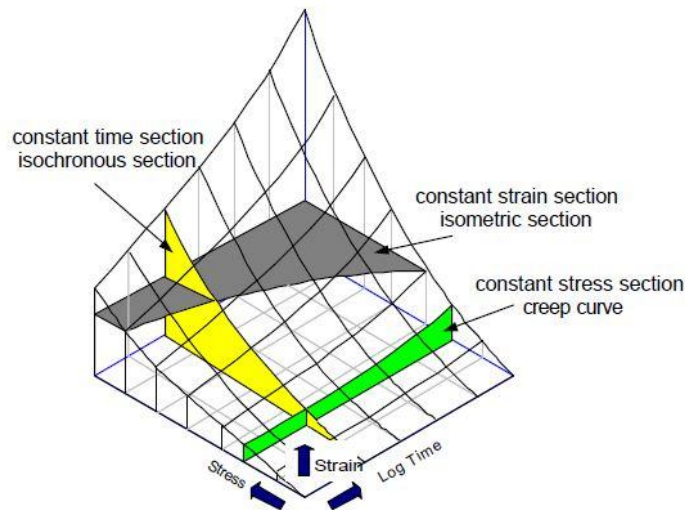


Figure 6. Constant stress-strain-time coordinates

3.3.1 Creep, constant load

When a plastic material is subjected to a constant load, it deforms continuously (Figure 7). The initial strain is roughly predicted by its stress-strain modulus. The material will continue to deform slowly with time indefinitely or until rupture or yielding causes failure. The primary region is the early stage of loading when the creep rate decreases rapidly with time. Then it reaches a steady state which is called the secondary creep stage followed by a rapid increase (tertiary stage) and fracture. This phenomenon of deformation under load with time is called creep. Of course, this is an idealized curve. Some materials do not have secondary stage, while tertiary creep only occurs at high stresses and for ductile materials.

All plastics creep to a certain extent. The degree of creep depends on several factors, such as type of plastic, magnitude of load, temperature and time.

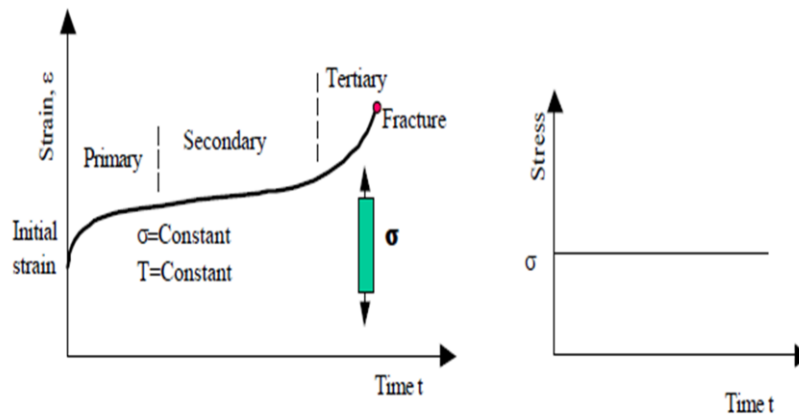


Figure 7. Creep curve for plastics, a constant load is applied

If the applied load is released before the creep rupture occurs, an immediate elastic recovery equal to the elastic deformation, followed by a period of slow recovery is observed (Figure 8). The material in most cases does not recover to the original shape and a permanent deformation remains. The magnitude of the permanent deformation depends on length of time, amount of stress applied, and temperature.

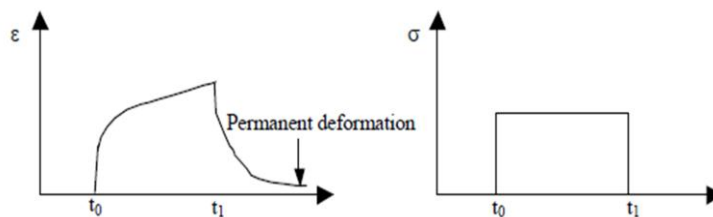


Figure 8. Creep curve with recovery. A constant load is applied at t_0 and removed at t_1

The creep rupture is basically similar to a creep test with the exception that it is continued until the material fails. Since higher loads are used, creep rates are higher and the material fails in a shorter time (usually terminated in 1000h). This test is useful in establishing a safe envelope inside which a creep test can be conducted. The basic information obtained from the stress rupture test is the time to failure at a given stress. Based on this data, a safe stress can be determined below which it is safe to operate, given the time requirement of the end use application. The construction of the creep rupture envelope is shown in Figure 9.

Test is conducted under constant stresses and the points of the onset of tertiary stage are connected to form the creep rupture envelope.

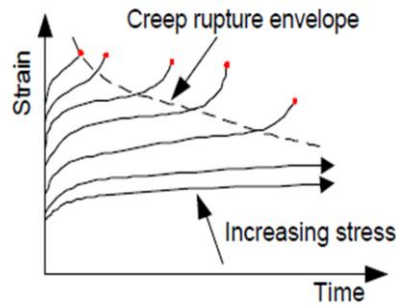


Figure 9. Creep rupture envelope

3.3.2 Stress relaxation, constant strain

Stress relaxation is defined as a gradual decrease in stress with time under a constant deformation or strain. This behavior of polymer is studied by applying a constant deformation to the specimen and measuring the stress required to maintain that strain as a function of time.

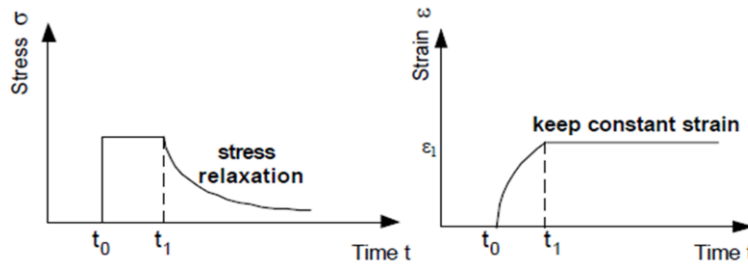


Figure10. Stress relaxation of plastics

Stress relaxation test can be used for some practical applications. For example, low stress relaxation is desired for threaded bottle closures. The stress data obtained from stress relaxation test can be used to calculate transient modulus for plastics design by simply dividing the stress at a particular time by the applied strain. The 3 dimensional stress-strain-time relationship can also be constructed by stress strain relaxation test as shown in Figure 10. However, stress relaxation test is more difficult to perform than creep test and has limited practical applications.

The following non-material parameters all affect stress relaxation in polymers :

- Magnitude of initial loading
- Speed of loading
- Temperature (isothermal vs non-isothermal conditions)
- Loading medium
- Friction and wear
- Long-term storage

3.3.3 Design with plastics

Design with plastics can be divided into two categories, design for strength and design for stiffness. The strength of a component is limited by the yield strength and rupture resistance of the material from which it is made. As shown in Figure 11, a creep rupture envelope can be obtained from creep test. For a expected life time, the maximum stress allowed (s_4) can be decided from the creep rupture envelope line. Design for stiffness with creep curves proceeds by establishing the maximum strain acceptable ϵ_{max} , thereby establishing a horizontal line on the creep diagram correspondingly. The expected lifetime t_L of the part is also determined, and the maximum stress permissible is found on the creep curve at the intersection of these two lines.

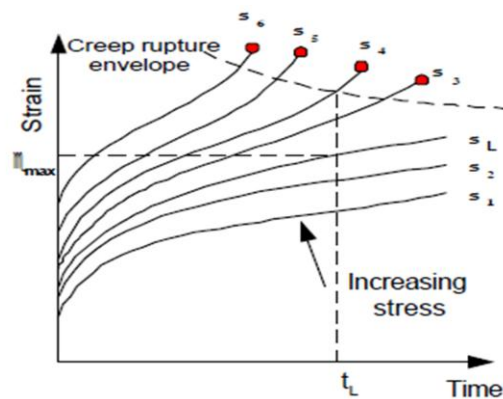


Figure 11. Design criteria by creep curves

As shown in Figure 10, many combinations of s and time will yield this maximum strain. For a desired lifetime t_L , however, there is one maximum level s_L which satisfies the maximum strain. Design basis selection depends on the specific application. Usually, strain or dimension requirement is more critical, and design for stiffness is favored in this case. If

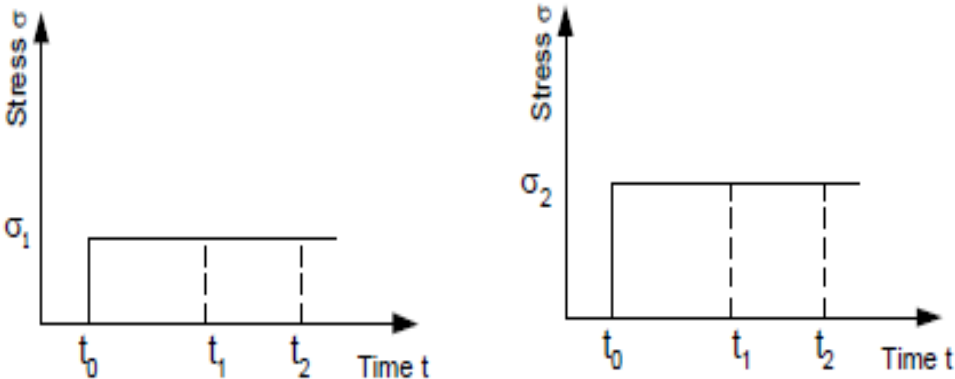
the dimension precision of the component under discussion is not so important compared as strength, design for strength is then used accordingly. For complicated structures, both can be used for design criteria to ensure successful material performance during service time.

Objective of creep

Since the pressure loads of the heat exchanger are nearly constant over time, creep behavior, i.e. strain under constant load, is the main consideration for our structure design. Aim is to get stress-strain-time relationship (constitutive law) and creep rupture data for our design basis. Since the service time is very long, 10 years, property prediction methods should be used for obtaining constitutive law. Time temperature superposition is one of the most important methods to predict long term properties using short duration tests. By creep test data in one or two months, creep behavior after some years can be predicted with acceptable accuracy.

3.3.4 Linear and nonlinear viscoelasticity

The time dependent material behavior is often referred to as **viscoelasticity**. If a constant load σ_1 is applied to a viscoelastic specimen, the time dependent strain is recorded as ϵ_1 as shown in Figure 12(a). After some period of time, the load is removed. Suppose the specimen is allowed to recover and a larger stress σ_2 is applied. The time dependence of the strain ϵ_2 is shown in Figure 12(b).



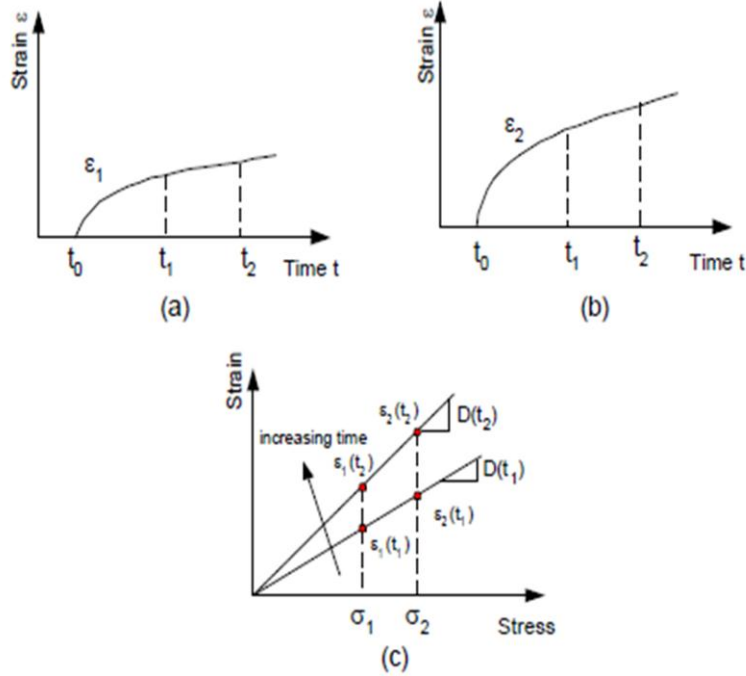


Figure 12. Linear viscoelastic creep: (a) constant stress σ_1 applied at time t_0 leads to time dependent strain $\epsilon_1(t)$; (b) a higher stress applied at t_0 leads to time dependent strain $\epsilon_2(t)$; (c) from (a) and (b) the strains at t_1 and t_2 are linear in the stress

If at a particular time t_1 and t_2 after loading, ϵ_1 and ϵ_2 are linear with the magnitude of correspondent stresses, σ_1 and σ_2 , the stress strain relationship can be given by

$$\frac{\epsilon_1(t_1)}{\sigma_1(t_1)} = \frac{\epsilon_2(t_1)}{\sigma_2(t_1)}, \frac{\epsilon_1(t_2)}{\sigma_1(t_2)} = \frac{\epsilon_2(t_2)}{\sigma_2(t_2)} \quad (3)$$

Thus, if at an arbitrary time t , the strains at the two stresses can be expressed as

$$\frac{\epsilon_1(t_1)}{\sigma_1(t_1)} = \frac{\epsilon_2(t_1)}{\sigma_2(t_1)}, \frac{\epsilon_1(t_2)}{\sigma_1(t_2)} = \frac{\epsilon_2(t_2)}{\sigma_2(t_2)} \quad (3)$$

$$\frac{\epsilon_1(t)}{\sigma_1(t)} = \frac{\epsilon_2(t)}{\sigma_2(t)} \quad (4)$$

The strains in the two experiments are proportional to the imposed stresses. In general, for stress σ , the creep compliance $D(t)$ can be given as the ratio of strain to stress at a certain time.

$$D(t) = \epsilon(t) / \sigma \quad (5)$$

This property is often characterized as linear viscoelasticity. In the linear range, the compliance is independent of stress, which means that the compliance is the same whether stresses used in the creep test are σ_1 , σ_2 or other stress levels.

A transition from linear to nonlinear viscoelasticity is shown in Figure 13

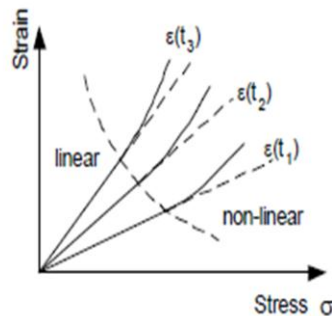


Figure 13. Linear-nonlinear transition of stress strain relationship with respect to different time levels
(Isochronous creep curve, data are taken from creep test at different stresses)

However, polymers generally exhibit linear viscoelastic property at low stresses such that the corresponding strain is below $\sim 0.5 \times 10^{-2}$. At higher stress levels, the material will assume nonlinear viscoelastic behaviors which will not obey the linear relation between stress and strain described by equation (4).

Since nonlinear behavior is very important to determine material behavior at moderate or higher stress levels, some models have been suggested for different kind of polymers.

3.3.5 Creep models

Although creep data are most accurately presented as the plot of strain vs. time for various stresses and temperatures, many theoretical and empirical relations have been suggested for the dependence of creep strain on stress and temperature for plastics. A simple model is a Kelvin (or Voigt) unit, which consists of a Hookean spring and a Newtonian dashpot as shown in Figure 14. The spring models the elastic response while the dashpot models the viscous (or time dependent) response to load.

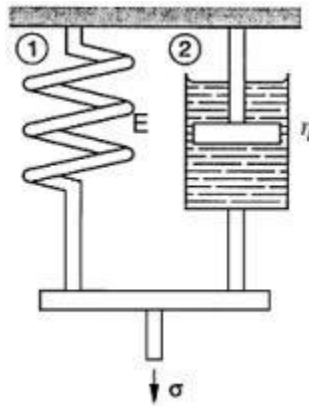


Figure 14. Schematic diagram of Kelvin model

The governing equation for Kelvin model is;

$$\sigma = E\varepsilon + \eta\dot{\varepsilon}, \quad (1)$$

E and η are the viscosity and Young's modulus respectively. Using the above equation, the strain in a creep test (constant stress) in the Kelvin model can be solved for as

$$\varepsilon(t) = \frac{\sigma_0}{E}(1 - e^{-t/\tau}), \quad \tau = \eta/E, \quad (2)$$

where τ is called the relaxation time at which the strain is 63.2% of σ_0/E .

Figure 15 shows the creep and recovery curve of Kelvin model. This is a twoelement-model. A large number of spring/dashpot components are usually needed to reasonably describe creep behavior over decades of time. These models can be used to construct constitutive law. And the unknown material constants, i.e. E and η can be curve fit from experimental data. Since different plastics show different creep properties, many models, which describe the relationship between stress, strain, time and temperature, have been suggested.

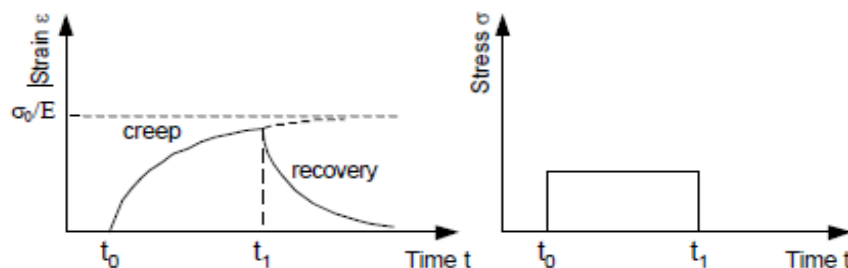


Figure 15. Creep and creep recovery response of Kelvin model

3.3.5 Superposition Principles

There are two superposition principles, which are important in predicting creep behavior of plastic materials under various test conditions. The first of these is the Boltzmann Superposition Principle, which describes the response of a material to different loading histories. The second is the Time Temperature Superposition Principle or the WLF equation, which describes the equivalence of time and temperature. The Boltzmann superposition principle states that the response of a material to a given load is independent of the response of the material to any load, which is already on the material. The deformation of a specimen is directly proportional to the applied stress, when all deformations are compared to equivalent times. It is only valid in linear viscoelastic region. For the case of creep, the total strain may be expressed by

$$\epsilon(t) = D(t - \tau_1)\sigma_1 + D(t - \tau_2)(\sigma_2 - \sigma_1) + \dots + D(t - \tau_i)(\sigma_i - \sigma_{i-1}) \quad (6)$$

Or

$$\epsilon(t) = \int_{-\infty}^t D(t - \tau) d\sigma(\tau) \quad (7)$$

where $D(t) = 1/E(t)$ is the compliance function, which is a characteristic of the polymer at a given temperature and initial stress. Figure 16 shows the creep curve by Boltzmann superposition principle.

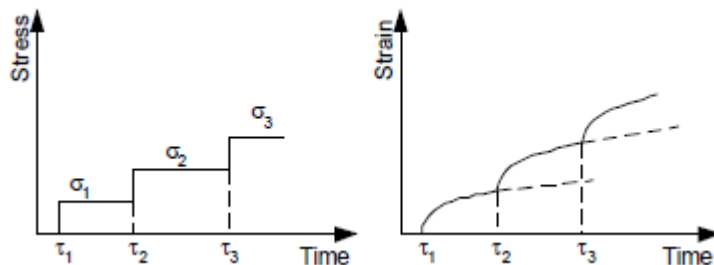


Figure 16. Boltzmann superposition principle

At time τ_1 , stress σ_1 is applied and the strain induced can be given by

$$\epsilon_1(t) = \sigma_1 D(t) \quad (8)$$

According to linear viscoelasticity, the compliance $D(t)$ is independent of stress, i.e. $D(t)$ is the same for all stresses at a particular time. If stress increment $\sigma_2 - \sigma_1$ is applied at time τ_2 and the strain increase due to stress increment $\sigma_2 - \sigma_1$ is

$$\varepsilon_2(t) = D(t - \tau_2)(\sigma_2 - \sigma_1) \quad (9)$$

Likewise, the strain increase due to $\sigma_3 - \sigma_2$ can be given by

$$\varepsilon_3(t) = D(t - \tau_3)(\sigma_3 - \sigma_2) \quad (10)$$

Further strains induced by stress increment which can be positive or negative, are added to give equation (6) or (7).

4. MATERIAL AND METHODS

4.1 Material

The material used in this investigation is a commercially available PTFE (DuPont Teflon_®), which is characterized by a density of $2.18 \times 10^3 \text{ kg/m}^3$ and a melting temperature about 327°C . The standard tension test specimens were manufactured from PTFE with shape and size specified by ASTM D-638-08 (Type I). The initial gage length l_0 and initial cross section A_0 are, respectively, 50 mm and 26 mm^2 (13 mm x 2 mm) as illustrated in Figure 17.

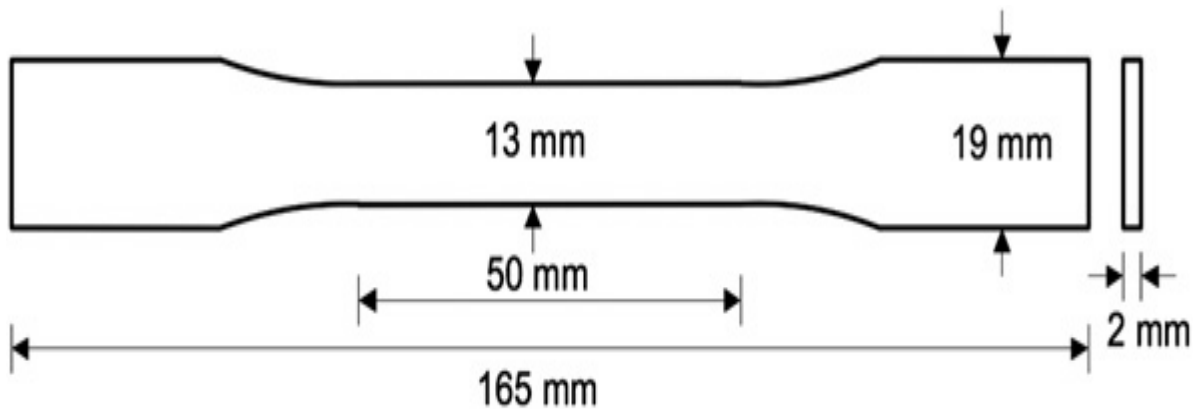


Figure 17. Standard tension test specimen dimensions.

4.2 Test procedure

Tensile tests were performed using an electromechanical Labortech, MPTest 5.50, sensing unit AST type KAF 5 kN, Czech Republic universal testing machine. This type of machine has two crossheads; one is adjusted for the length of the specimen and the other is driven to apply tension to the test specimen.

There are two types: hydraulic powered and electromagnetically powered machines. The machine must have the proper capabilities for the test specimen being tested. There are four main parameters: force capacity, speed, and precision and accuracy. Force capacity refers to the fact that the machine must be able to generate enough force to fracture the specimen. The machine must be able to apply the force quickly or slowly enough to properly mimic the actual application. Finally, the machine must be able to accurately and precisely measure the gauge length and forces applied. During this process, the elongation of the gauge section is recorded against the applied force. The data is manipulated so that it is not specific to the geometry of the test sample. The elongation measurement is used to calculate the engineering strain, ϵ , using the following equation:

$$\varepsilon = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0}$$

where ΔL is the change in gauge length, L_0 is the initial gauge length, and L is the final length. The force measurement is used to calculate the engineering stress, σ , using the following equation:

$$\sigma = \frac{F_n}{A}$$

where F is the tensile force and A is the nominal cross-section of the specimen. The machine does these calculations as the force increases, so that the data points can be graphed into a stress–strain curve. It is important to emphasize that all experiments were carried out at room temperature, i.e. 25°C. The engineering strain rates ($e'1 = 7,8 \text{ mm/s}$, $e'2 = 20 \text{ mm/s}$, $e'3 = 27 \text{ mm/s}$), the constant stress (creep) approximately at 75N and constant strain (stress relaxation) at 0.00035 were controlled by using the measured elongation as input to the control system of the testing machine.

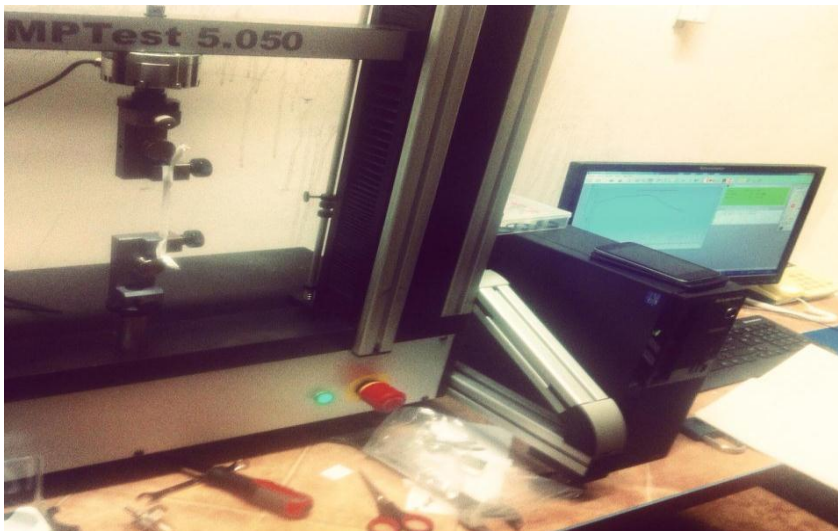


Figure 18. Labortech, MPTest 5.50, sensing unit AST type KAF 5 kN, Czech Republic universal testing machine.



Figure 19. Prepared PTFE test sample
- 50 mm and 26 mm²(13 mm x 2 mm)

6.RESULTS AND DISCUSSION

True and engineering relationship definitions

Using prescribed elongation $\Delta l(t)$ and applied force $F(t)$ experimentally measured, in addition to the initial gage length l_0 and initial cross section A_0 , the engineering strain e and the engineering stress s are given by

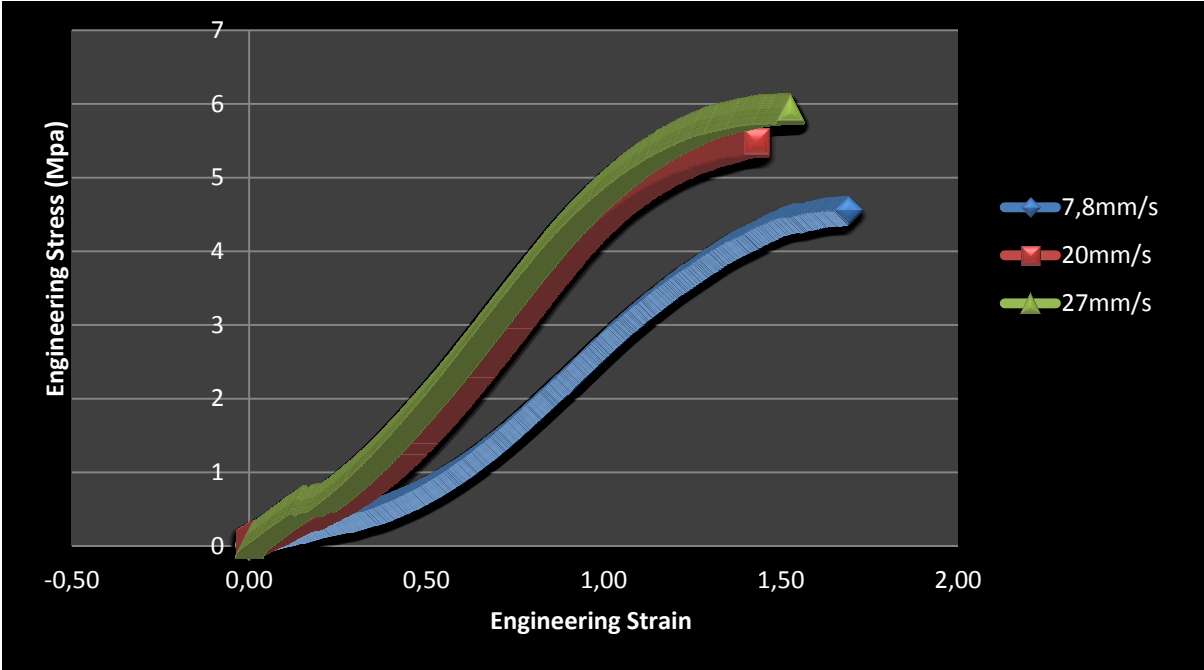


Figure 20. Engineering Strain-Stress Curves

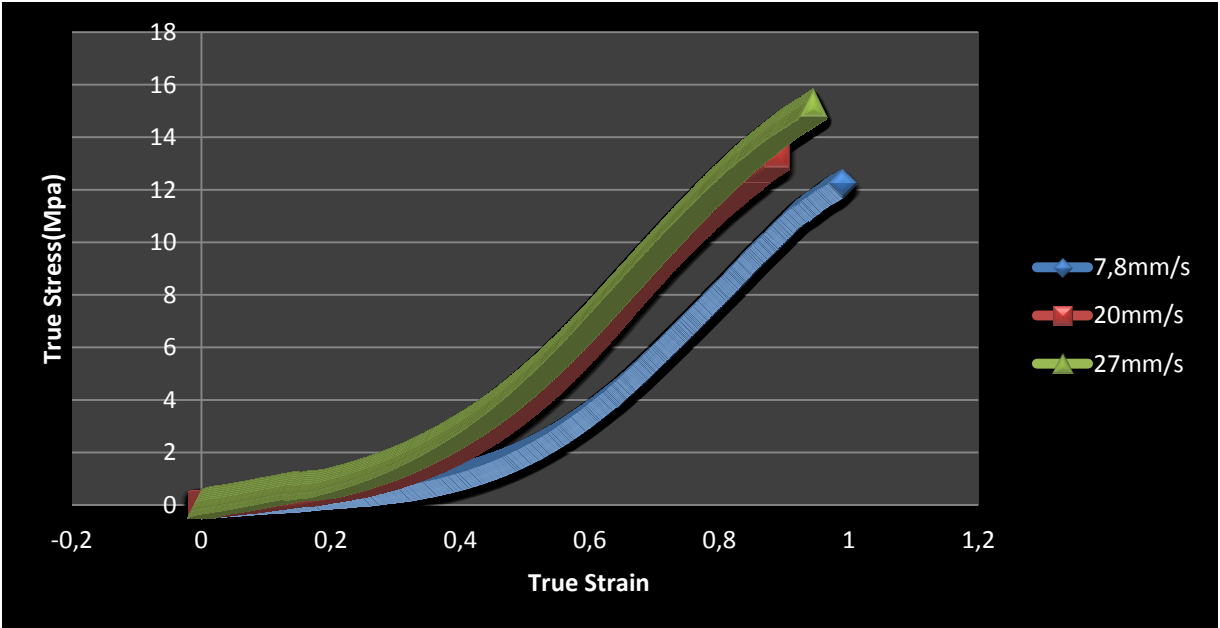


Figure 21. True Strain-Stress Curves

$$e(t) = \Delta l(t)/l_0 \text{ and } s(t) = F(t)/A_0 \quad (1)$$

With constant volume, the true strain and true stress as a function of e and s are defined as

$$\epsilon = \ln(1+e) \text{ and } \sigma = s(1+e) \quad (2)$$

Figs. 20 and 21 illustrate engineering and true stress-strain curves of PTFE for different constant engineering strain rates: $\dot{e}'1 = 7,8 \text{ mm/s}$, $\dot{e}'2 = 20 \text{ mm/s}$, $\dot{e}'3 = 27 \text{ mm/s}$. These results were obtained from experimental data using Eqs. (1) and (2). The curves show a strong strain rate dependency, in which the flow stress increases with strain rate.

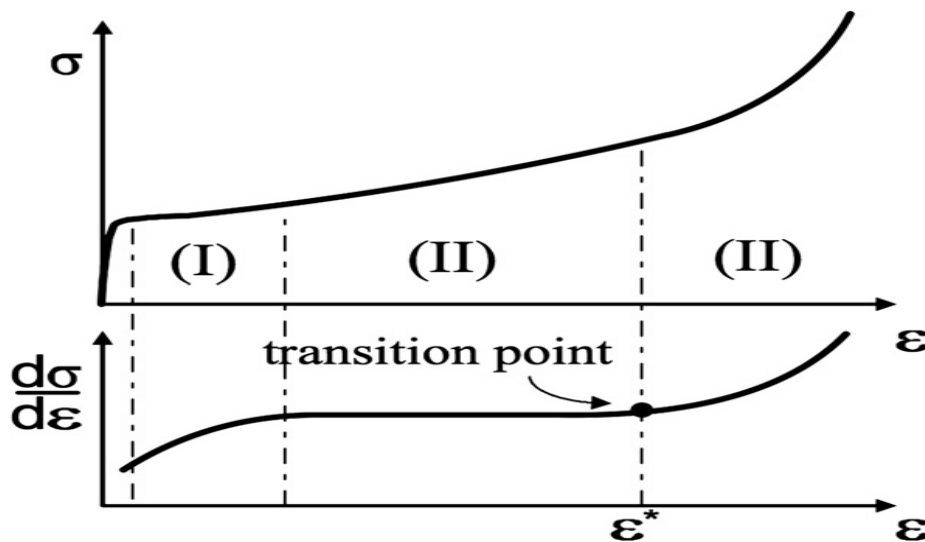


Figure 22. Schematic representation of stress-strain and slope curves: definition of ϵ^*

Disregarding the small strain region ($\epsilon < 4\%$), for all cases, the true stress-strain curve has three distinct regions: (i) region I, initially σ/ϵ is high, however it gradually decreases; (ii) region II, where σ/ϵ tends to zero and (iii) region III, σ/ϵ increases again (see Fig. 22). Fig. 22 illustrates a schematic representation of the true stress-strain curve and the slope of this curve, in which ϵ^* is defined as the strain value associated with the transition between region II and region III. Experimentally, it is observed that ϵ^* is not very sensitive to the strain rate and, thus, it can be assumed as a material constant.

One of the characteristics of PTFE is to present large deformations under tension prior to local failure. Fig. 23 illustrates a sequence of images of a typical PTFE specimen under monotonic loading until failure. Specimen deformation is very homogeneous. The high strain rate sensitivity of flow stress confers a high resistance to neck development: the specimen gets thinner in a very uniform manner, rather than forming a 'neck'. In general, failure is localized

and characterized by a crack perpendicular to the tensile axis. Under slow extension conditions a neck was not formed locally but was propagated through the entire specimen as a result of hardening of the material.



Figure 23. Images of PTFE specimen under monotonic loading until failure.

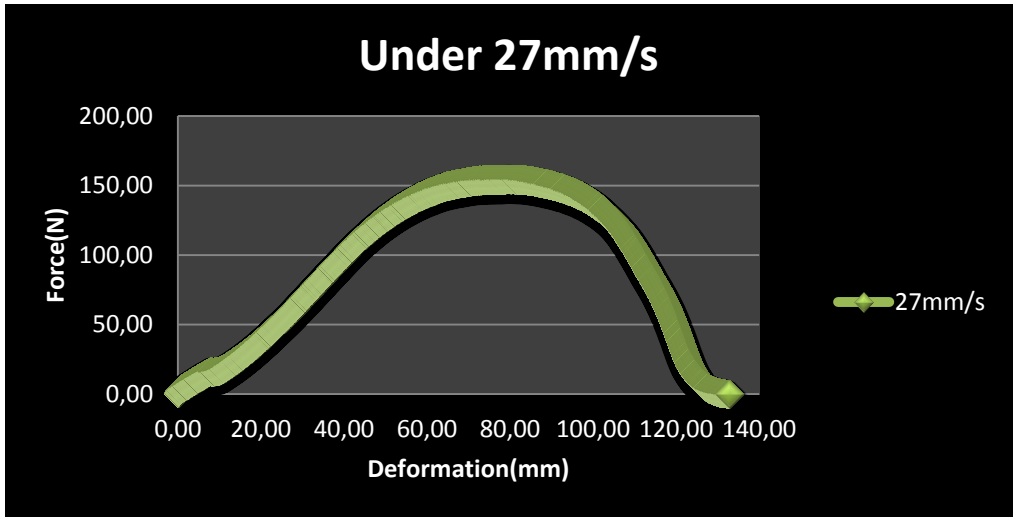
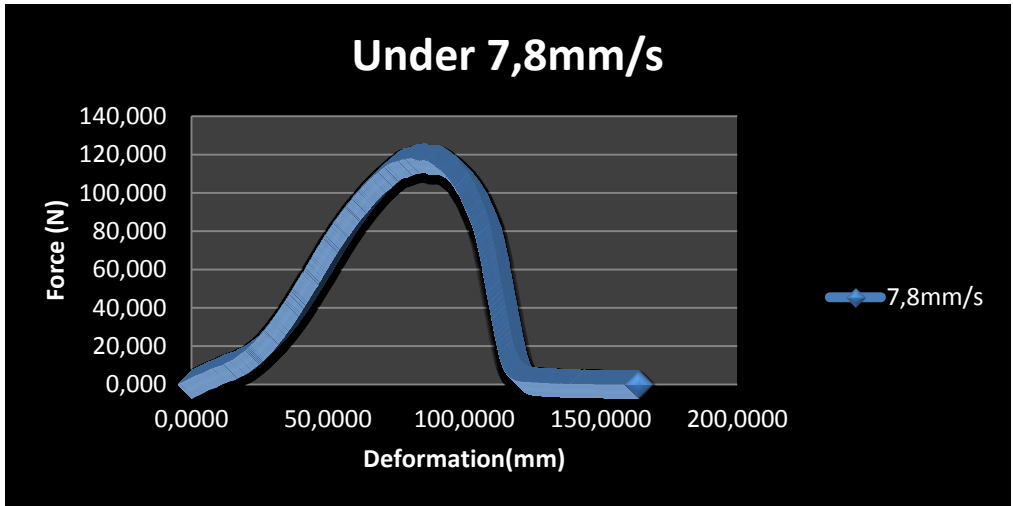


Figure 24-25-26. Force&Deformation Curves for different strain rates

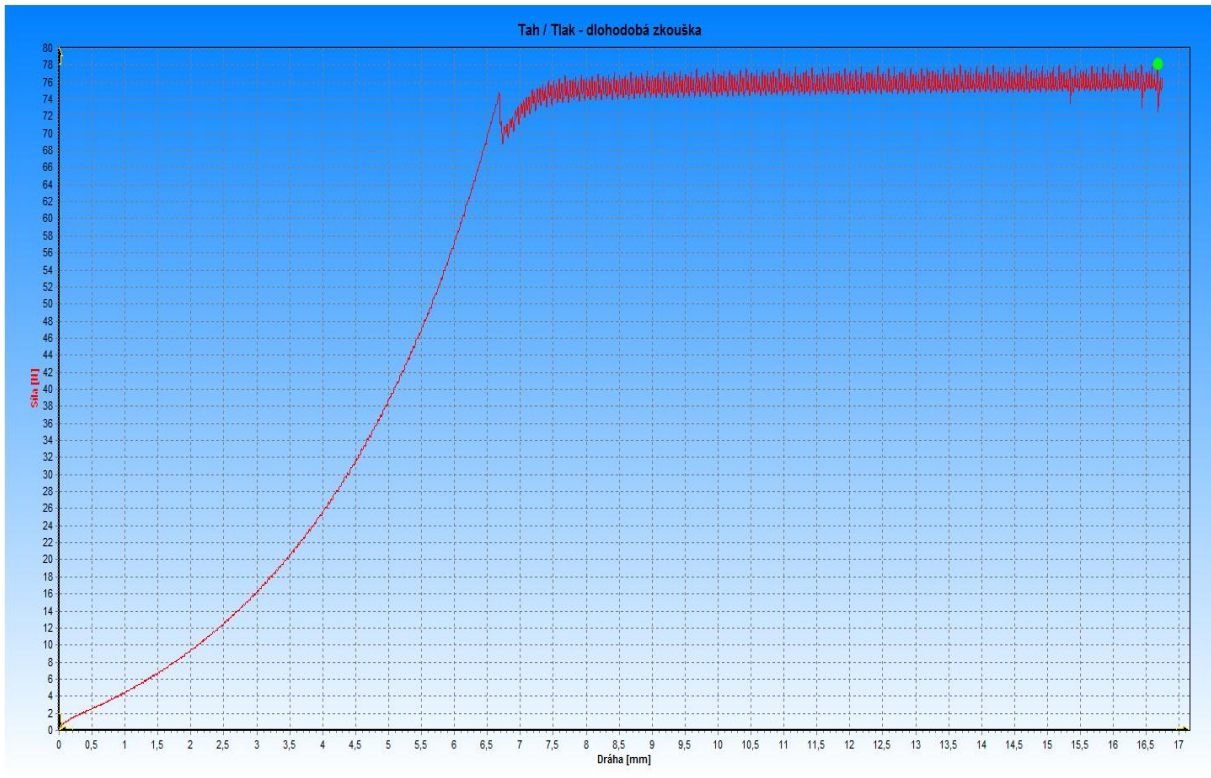


Figure 27. Force&Deformation Curve (Creep Test)

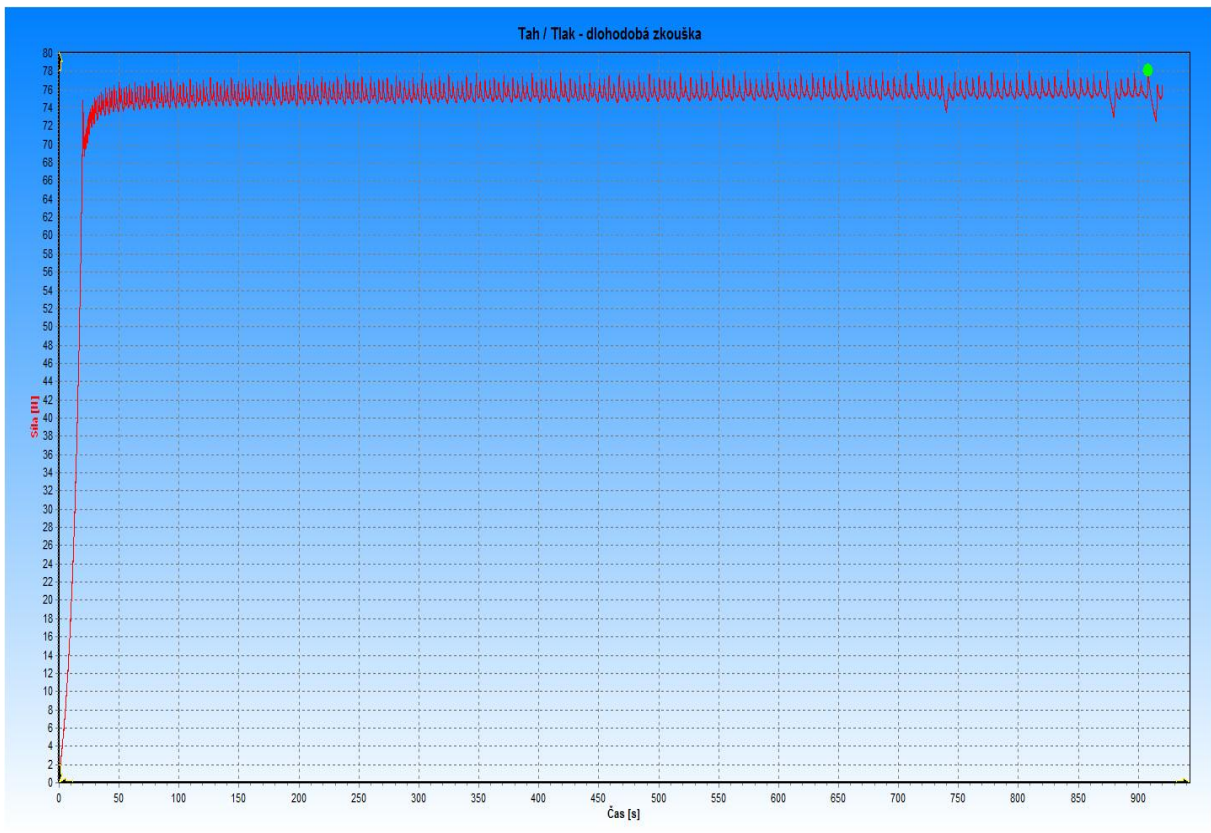


Figure 28. Force&Time Curve (Creep Test)

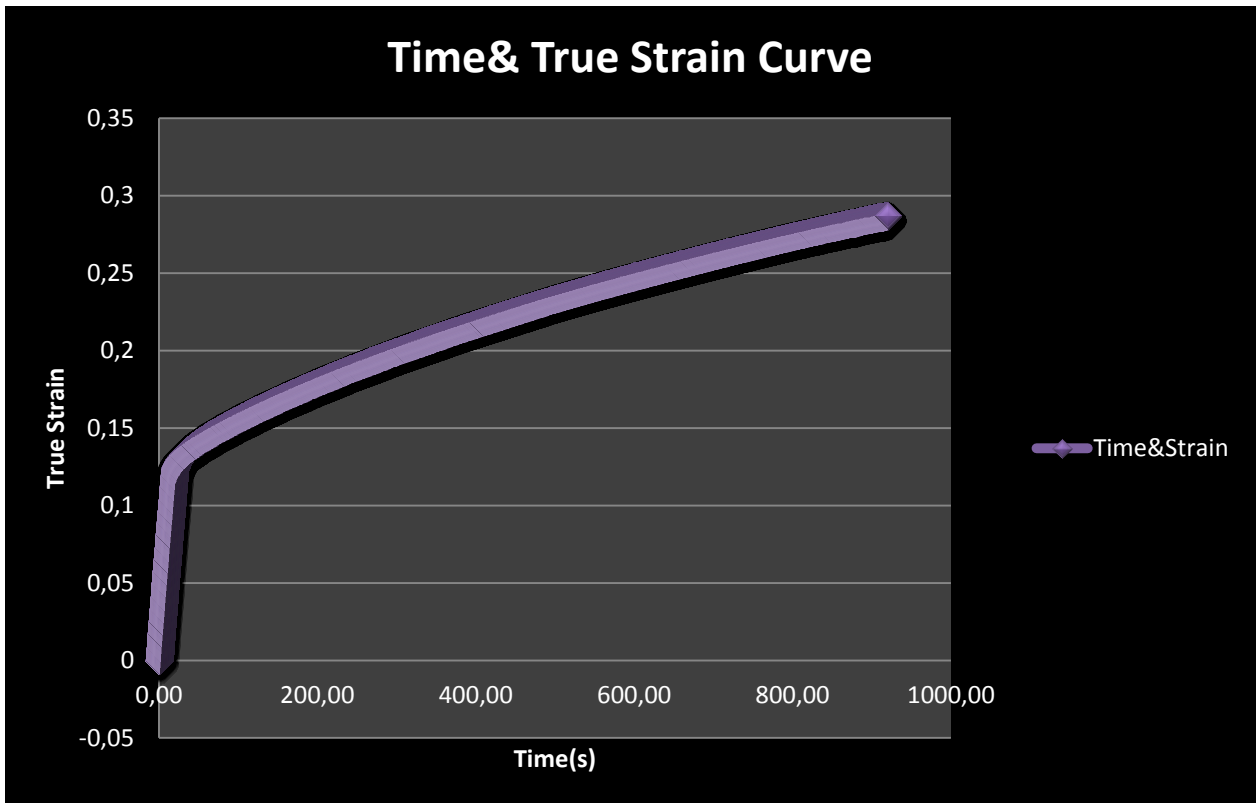


Figure 29. Time&Strain Curve (Creep Test)

Creep resistance can be defined as a material's ability to resist any kind of distortion when under a load over an extended period of time. For optimum performance and maximum lifetime, engineering plastics, which are subjected to long-term loading, should have a high creep resistance (i.e. low plastic deformation under load). Figure 27, 28 and 29 illustrate creep graphs approximately at approximately 75N constant load. Initial strain or deformation occurs instantaneously as a load is applied to PTFE resins. Following this initial strain is a period during which the part continues to deform but at a decreasing rate.

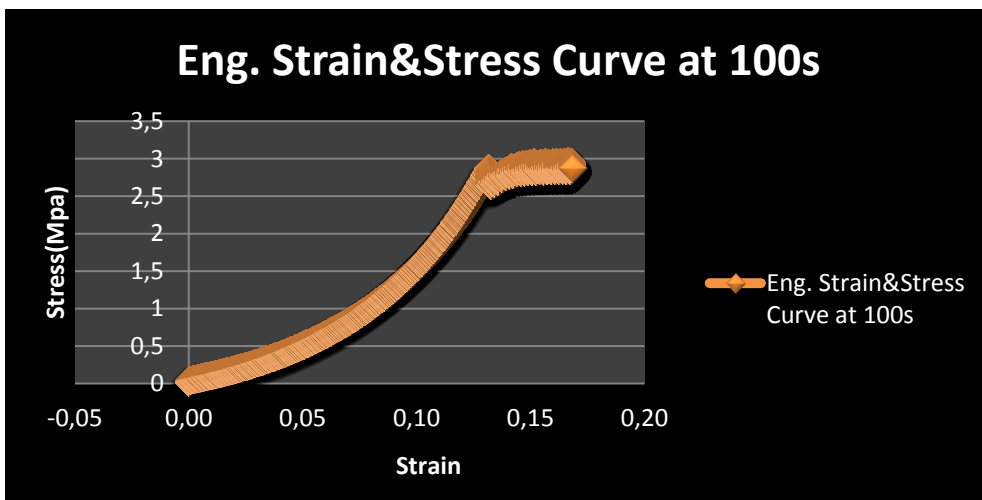


Figure 30. Eng. Stress&Strain Curve at Creep around 100. second

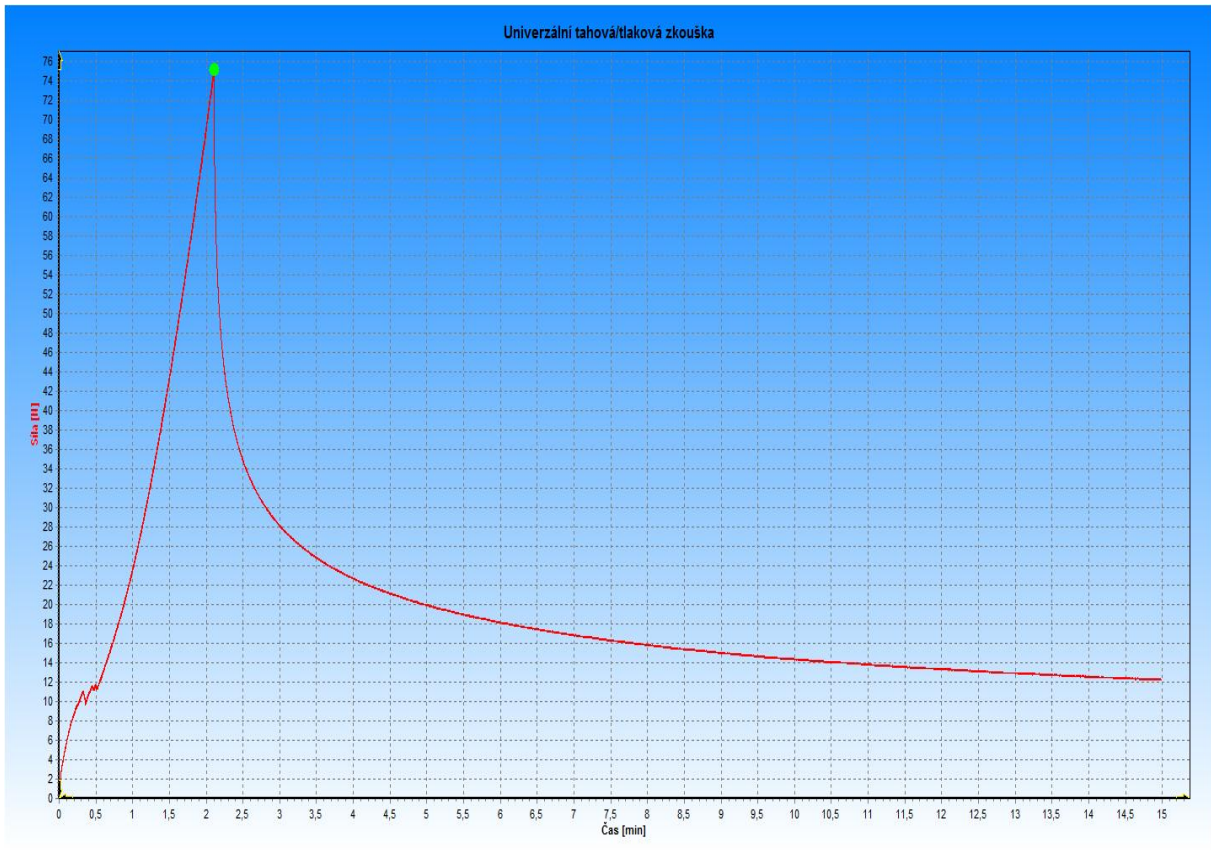


Figure 31. Force&time curve at constant deformation (relaxation test)

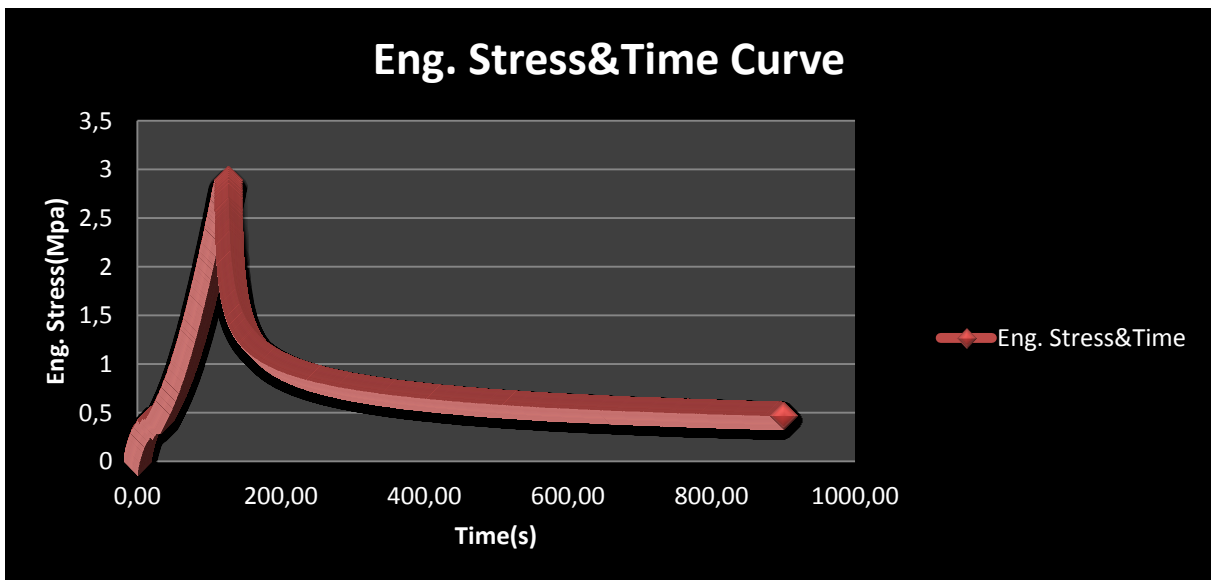


Figure 32. Eng. Stress&Time Curve(Relaxation Test)

The bonds of PTFE within each chain are strong covalent bonds. The secondary bonds that act between two chains are weaker than the covalent bonds, and the larger the distance between two chains, the weaker the secondary bond. In a crystalline region the chains are tightly packed and consequently, the secondary bonds are stronger than in an amorphous

region. The degree of crystallization will therefore affect the strength of the polymer. By adding energy, e.g. by rising the temperature, the distance between the chains will increase and hence, the material will soften. Similarly, the distance between two chains will increase upon stretching the material. However, the chains in the stretched material will slide with time, causing the applied stress to decrease. This phenomenon is known as stress relaxation and is due to the viscoelastic behavior of polymers. At Figure 30 curves for tensile stress relaxation illustrate the rates at which tensile stress decays when the specimen is maintained at 0,00035 constant strain.

For future works, some ideas have been thought with taking into account past literature to understand mechanical behaviours of PTFE better, we can describe the response of a material to different loading histories at creep tests to understand nonlinear viscoelastic behaviour better with commenting isochronous creep curves , and we can make comment about stiffness of PTFE better with evaluating envelope strain-time curve for this test, we can get life time of the PTFE and we can find maximum stress permissible from intersection points...

And we know that PTFE is a complex material. It exhibits pressure and temperature generated phase transitions, is semi-crystalline and is insoluble in all common solvents . So it will be nice to work on temperature-dependent behaviours to understand better mechanical behaviours of PTFE on these tests which we made. Being chemically inert makes characterization difficult. Methods to estimate crystallinity ,molecular weight and crystallographic morphology. we can also compare % variation in crystallinity of different products of PTFE. The crystalline percentage of PTFE may be altered for a given molecular weight by altering the heat treatment profile used in production (thermal history). In order to understand some of the results, production of some PTFE with a different level of crystallinity was useful.

It can be also very beneficial to see mechanical behaviours of PTFE under compression test for different strain rates and creep test, A comparison of uniaxial tensile and compressive creep isochronol data can help us to understand results better.

7.CONCLUSION

We observed that the stress-strain & time relationship by loading PTFE specimen with constant stress (creep) and constant strain (relaxation) with using extensometer. Primary creep started at a rapid rate and got slow with time. Then it had a relatively uniform rate. The disproportionately greater increase of strain for equal stress increments confirms that PTFE is not a linear viscoelastic material in tension. The slope of the line around the 0.15 strain region indicates that in tension the material is approaching instability. We can notice that the non-linear viscoelastic behavior of the material extends into the low strain region. And the stress relaxation was most significant right after loading, but it continued for long time due to slipping of the molecular chains. The strain relaxation curve shows us the time dependent decrease of the measured stress. We can notice that PTFE has poor resistance to creep and stress relaxation.

The strain rate dependency of polytetrafluoroethylene (PTFE) specimens in tensile tests was analyzed. The large deformations were measured with using universal extensometer. We observed that strain rate sensitivity of flow stress that confers a high resistance to neck development and results in the high tensile elongations characteristic of superplastic behavior of PTFE. Specimens get thinner in a very uniform manner, rather than forming a neck and then fractured. The strain- rate stress curves show a strong strain dependency, in which the flow stress increases with strain rate. So we can say that the stress response in the uniaxial tension tests shows a nonlinear strain-stress behaviour depending on strain rate.

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Enclosures

Figure 1. Structure of PTFE

Figure 2. The variation of the linear thermal expansion coefficient with temperature

Figure 3.(Stress-Strain Curve)

Figure 4. Ductility&Brittle comparison

Figure 5. Figure to illustrate the relationship between S and SR. If we assume that this deformation (in this case the shortening) takes 2 seconds, the strain rate is equal to $-0,2/2$ sec, which is $-0,1s^{-1}$.

Figure 6. Constant stress-strain-time coordinates

Figure 7. Creep curve for plastics, a constant load is applied

Figure 8. Creep curve with recovery. A constant load is applied at t_0 and removed at t_1

Figure 9. Creep rupture envelope

Figure10. Stress relaxation of plastics

Figure 11. Design criteria by creep curves

Figure 12. Linear viscoelastic creep: (a) constant stress σ_1 applied at time t_0 leads to time dependent strain $\epsilon_1(t)$; (b) a higher stress applied at t_0 leads to time dependent strain $\epsilon_2(t)$; (c) from (a) and (b) the strains at t_1 and t_2 are linear in the stress

Figure 13. Linear-nonlinear transition of stress strain relationship with respect to different time levels (Isochronous creep curve, data are taken from creep test at different stresses)

Figure 14. Schematic diagram of Kelvin model

Figure 15. Creep and creep recovery response of Kelvin model

Figure 16. Boltzmann superposition principle

Figure 17. Standard tension test specimen dimensions.

Figure 18. Labortech, MPTest 5.50, sensing unit AST type KAF 5 kN, Czech Republic universal testing machine.

Figure 19. Prepared PTFE test sample- 50 mm and 26 mm²(13 mm x 2 mm)

Figure 20. Engineering Strain-Stress Curves

Figure 21. True Strain-Stress Curves

Figure 22. Schematic representation of stress-strain and slope curves: definition of ϵ^*

Figure 23. Images of PTFE specimen under monotonic loading until failure.

Figure 24-25-26. Force&Deformation Curves for different strain rates

Figure 27. Force&Deformation Curve (Creep Test)

Figure 28. Force&Time Curve (Creep Test)

Figure 29. Time&Strain Curve (Creep Test)

Figure 30. Eng. Stress&Strain Curve at Creep around 100. second

Figure 31. Force&time curve at constant deformation (relaxation test)

Figure 32. Eng. Stress&Time Curve(Relaxation Test)